PROJECT NUMBER: 1754
PROJECT TITLE : Spectroscopic Studies of Tobacco and Smoke Components
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NMR

Two 2,4-disubstituted butyrolactones were analyzed for Ken Podraza to determine the cis/trans configuration by using chemical shift and coupling constant data. A kinetic study of the rhamnose-ammonia reaction showed that the reaction is 75-80% complete after 18 hours and complete after 72 hours (no rhamnose detected). Several serricornin and anhydroserricornin samples were analzyed to determine isomer ratios for the diastereoisomers.[1]

The carbonyl/carboxyl region of the ^{13}C CP/MAS spectra of tobacco is very distinctive for different tobacco samples. The spectra of several model compounds were obtained, including citric acid trisodium salt dihydrate, citric acid monosodium salt (anhydrous), and malic acid disodium salt dihydrate. The sharp 180 ppm resonance in burley tobacco may be due to the monosodium (or some other cation) salt of citric acid since it gives a sharp resonance at 180 ppm and a weaker signal at 173 (which would overlap with the uronic acid carboxyls in tobacco spectra). The free citric acid or the trisodium salt gives three resolved signals which do not resemble the tobacco signals. The malic acid salt seems to be ruled out since the carboxyl signals are shifted slightly downfield to 182 & 183 ppm. The two malic carboxyl signals were found to have different cross polarization efficiencies and rotating reference frame relaxation times.[5]

^{13}C CP/MAS spectra were also obtained on pansy flowers, a whole freeze-dried tobacco cell suspension culture, and several carbonate salts.[5]

MS

A new 160 M byte disk was installed on the SS 200 data system and working files were transferred to it. A head crash occurred on the 30 M byte disk, causing a loss of our libraries. We were able to recover about 70% of the data, and we are in the process of rebuilding the libraries. Additional problems with our magnetic tape unit has also caused a slow down in recovery from these data system problems.[3]

Four rhamnosylamine samples were analyzed for G. Chan using DCI/MS. Each was found to be pure.[2]

Several compounds were evaluated for use as FAB calibration standards. A perfluoro compound, Ultramark 2500, was found to be excellent for negative FAB, since its mass spectrum contains abundant ions at masses from 50 to 1000 at intervals of about 50u. Additional data have been obtained by FAB for tobacco glycosides and for a tobacco leaf extract fraction isolated by HPLC by Bob Armstrong. In general, the glycosides show strong M + 1 ions, and ions corresponding to loss of the saccharide units. They also readily form sodium and potassium adduct ions in the positive mode.[2]

ELECTRONICS[4]

A problem in a printed circuit board was identified and corrected on the Nermag MS, thus avoiding a downtime of several weeks. The gas cell on the P. E. 221 IR was overhauled and realigned, thus improving signal transmission from 30% to 55%. Repairs were made on other various IR instruments and a digital voltmeter was constructed and installed on the MAT 112S MS to provide a digital mass display.

A considerable amount of time was spent in efforts to correct computer problems on the Bruker NMR. Replacement printed circuit boards have been ordered.

REFERENCES

1. R. Bassfield, N.B. 7398.
2. W. N. Einolf, N.B. 8040.
3. D. F. Magin, N.B. 7936.
4. S. C. Marrs
5. J. B. Wooten, N.B. 7826.